High Yield and Economical Production of Rare Earth Elements from Coal Ash

DOE Contract DE-FE0027167 – Phase 2

Physical Sciences Inc., Andover, MA, Center for Applied Energy Research, Lexington, KY
Winner Water Services, LLC, Sharon, PA

Presentation to:
Rare Earth Elements (REE) Program Portfolio,
2019 Annual Review Meeting, Pittsburgh, PA,
9-11 April 2019

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Presentation Outline

- Phase 2 Programmatic
- Rare Earths Recovery Process Overview
- Phase 2 Tasks, Schedule, Milestones, and Deliverables
- Summary of Phase 2 Results to Date
Phase 2 Programmatic

- **Area Of Interest (AOI) 2 program: Pilot Scale Technology**
  - Existing separation technology previously demonstrated successfully on bench scale
  - Ready or near-ready for design at pilot scale
    - Pilot plant design to be delivered at end of Phase I
  - Ready for scale up to commercial scale (design) at completion of Phase II

- **30-month Phase 2 program: 9/29/2017 – 9/31/2020**
  - Phase 1 ended 8/31/2017

- **Team:**
  - Physical Sciences Inc. (PSI), Andover, MA
  - Center for Applied Energy Research (CAER), Lexington, KY
  - Winner Water Services, LLC (WWS), Sharon, PA

- **Total Contract Value ~$7.5M = $6M DOE funds + $1.5M Cost Share**
Rare Earths Recovery Process Overview
Rare Earths Recovery Process Overview

- Physical separation stage, followed by a chemical separation stage, followed by a post-processing stage
- **Proposed Product:** REYSc-enriched mixture (dry concentrate)
- **Higher Value Products:** REY-rich & Scandium-rich concentrates
- **By-products:** Cement substitute, cenospheres, secondary fuel carbon...

REE = Rare Earth Elements
REY = REE + Yttrium
REYSc = REY + Sc
Post-Processing for REY and Sc Relative Content

- Post-processing leads to:
  - 3X increase in REY enrichment
  - Efficient separation from other contaminants
  - Product well-suited for REY separation
Phase 2 Overview
Overall Project Goal

• Develop and demonstrate a pilot scale plant to economically produce salable rare earth element-rich concentrates, including yttrium, scandium, and commercially viable co-products from coal ash feedstock; using environmentally safe, and high-yield physical and chemical enrichment processes.

• Phase 2 Project Metrics

<table>
<thead>
<tr>
<th>Performance Parameter</th>
<th>Threshold Value</th>
<th>Objective Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feedstock REYSc[^\d]* Content</td>
<td>&gt;300 ppm (Whole Mass Basis)</td>
<td>&gt;500 ppm (Whole Mass Basis)</td>
</tr>
<tr>
<td>Total REYSc Enrichment in Final Concentrate</td>
<td>&gt;10 wt% (Elemental Basis)</td>
<td>&gt;20 wt% (Elemental Basis)</td>
</tr>
<tr>
<td>Return on Investment[^*\d]*</td>
<td>&lt;7 y</td>
<td>&lt;5 y</td>
</tr>
<tr>
<td>Delivered Concentrate Quantity[^&amp;\d]*</td>
<td>~50 g[^$\d]*</td>
<td>~0.5 kg[^$\d]*</td>
</tr>
</tbody>
</table>

REYSc = Rare Earth Elements Plus Yttrium and Scandium, *Scale-dependent ~ 600 tpd, &Ten 5g split samples, 5g split sample required per solicitation.
Phase II Team

- The CAER, PSI, WWS team provides a complete integrated science, technology, engineering, technology transition, and commercialization solution for DOE/NELT

- Key Personnel:
  - **PSI:**
    - Dr. Prakash Joshi, PI/PM
    - Dr. Dorin Preda, Lead Chemist
    - Dr. David Gamliel, Lead Chemical Engineer/Process Modeling/TEA
  - **CAER:**
    - Dr. James Hower, Coal geochemistry, materials characterization
    - Dr. John Groppo, Mineral processing, feedstock logistics, site qualification
  - **WWS:**
    - Mr. Todd Beers: Chemical Engineering and technology commercialization
    - Mr. Michael Schrock, Chemical Engineering; Plant Design
Phase 2 Scope – I
Overall Program

- Demonstrate the Phase 1 REYSc separation/enrichment technology in pilot plant(s) with *decoupled* operating capacities of ~ 0.4 tpd physical processing, and ~ 0.5 tpd chemical processing.
  - Both pilot designs will be *modular* and *transportable*

<table>
<thead>
<tr>
<th>Phase</th>
<th>Start TRL</th>
<th>End TRL</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td>2 (Current)</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>2 (End)</td>
<td>5</td>
<td>6+</td>
</tr>
</tbody>
</table>
Phase 2 Scope – II

Physical Processing Pilot

• The Physical pilot plant comprises existing equipment at CAER facility in Lexington, KY, which was repaired/refurbished for Phase 2.

• The physical pilot is decoupled from the chemical pilot located at the WWS facility in Sharon, PA.
  – ~20 tons of selected ash transported to CAER facility.

• Physical processing pilot, will be operated over <~4 weeks for a significant demonstration while conserving project funds, producing 10 to 20 tons of the chemically processable ash fraction.

• Selected chemical processing operations may be collocated with physical processing to minimize amount of material to be processed in the chemical (pilot) plant, and to save transportation costs.

• Selected ash fraction to be transported to the WWS plant in Sharon, PA for chemical processing.
Phase 2 Scope – III
Chemical Processing Pilot

- Chemical processing pilot utilizes WWS’s existing, proven solvent extraction equipment to minimize costs, with additional equipment procured for pre-extraction operations and for reagent compatibility.

- The chemical pilot will be operated over ~ 2 to 4 weeks for a significant demonstration while also conserving project funds, producing ~ 50 to 500 grams of REYSc-enriched product deliverable.

- Environmentally safe disposal of waste products.

- Pilot will demonstrate the high recovery/recycling of reagents; > 95%.
Phase 2 Scope – IV
Chemical Micropilot & Techno-Economic Modeling

- A ~1-5 kg/d **Micropilot Plant** has been developed at PSI, Andover, MA
  - Quick turnaround validation of pilot plant processing parameters and to provide data for chemical pilot plant design

- A high fidelity **Aspen-based Techno-Economic Model** of REYSc recovery from coal ash using the results from physical and chemical pilot plant operations
  - Refine, enhance, and validate the Phase 1 model
  - Fidelity of the resulting Phase 2 model to be AACE Class 2
  - Model will be used to develop design of a commercial scale plant for profitable (ROI < 5-7 y) REYSc at the conclusion of Phase 2
Phase 2 Tasks, Schedule, Milestones, and Deliverables
# Phase 2 Schedule

<table>
<thead>
<tr>
<th>Task</th>
<th>Contract Month</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.0 Program Management</td>
<td></td>
</tr>
<tr>
<td>2.0 Site Host Agreement</td>
<td></td>
</tr>
<tr>
<td>3.0 Provide Split Samples</td>
<td></td>
</tr>
<tr>
<td>4.0 Physical Processing &amp; Ash Characterization</td>
<td></td>
</tr>
<tr>
<td>4.1 Physical Processing Pilot Developments</td>
<td></td>
</tr>
<tr>
<td>4.1.1 Physical Processing Plant Preparations</td>
<td></td>
</tr>
<tr>
<td>4.1.2 Physical Processing Plant Demonstration</td>
<td></td>
</tr>
<tr>
<td>4.2 Ash Fractions Characterization</td>
<td></td>
</tr>
<tr>
<td>5.0 Chemical Processing</td>
<td></td>
</tr>
<tr>
<td>5.1 Micropilot Plant Developments</td>
<td></td>
</tr>
<tr>
<td>5.2 Chemical Processing Plant Developments</td>
<td></td>
</tr>
<tr>
<td>5.2.1 Chemical Processing Plant Design</td>
<td></td>
</tr>
<tr>
<td>5.2.2 Chemical Processing Plant Construction/Installation</td>
<td></td>
</tr>
<tr>
<td>5.2.3 Chemical Processing Plant Shakedown</td>
<td></td>
</tr>
<tr>
<td>5.2.4 Chemical Processing Plant Optimization and Demo.</td>
<td></td>
</tr>
<tr>
<td>6.0 Techno-economic Modeling</td>
<td></td>
</tr>
<tr>
<td>7.0 Commercial Plant-Design</td>
<td></td>
</tr>
<tr>
<td>8.0 Engineering Development Support (Cost Share)</td>
<td></td>
</tr>
</tbody>
</table>

## Milestones / Reviews

- Kickoff @ NETL
- TIM #1 @ CAER
- Design Review @ NETL
- Prep. TIM #2 Site Visit @ CAER
- TIM #3 @ PSI
- TIM #4 @ WWS
- TIM #5 Readiness Review @ WWS
- TIM #7 @ WWS Chem Plant Demo.
- Final Brief @ NETL

## Deliverables

- Update Proj Plan
- Host Site Agreement
- Sys Testing, Ops, Sampling Plan for Physical Pilot
- Sys Testing, Sampling & Analysis Plan for Chemical Pilot
- Physical Pilot Test Report
- Chemical Pilot Shakedown Report
- Final REE Product Split Sample Delivery
- Chemical Pilot Test Rpt
- Techon Econ Analysis Rpt
- Tech Dev & Commercialization Plan

## Reports and Briefings

- Qtrly. #1
- #2
- #3
- #4
- #5
- #6
- #7
- #8
- #9
- Ph. 2 Final Report

TIM = Technical Interchange Meeting
# Phase 2 Milestones and Deliverables

<table>
<thead>
<tr>
<th>Milestone</th>
<th>Program Month</th>
<th>Planned Completion Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kickoff Meeting</td>
<td>1</td>
<td>24 January 2018</td>
</tr>
<tr>
<td>Updated Project Management Plan</td>
<td>1</td>
<td>31 January 2018</td>
</tr>
<tr>
<td>Technical Interchange Meeting (TIM) #1 @ CAER</td>
<td>3</td>
<td>15 March 2018</td>
</tr>
<tr>
<td>Quarterly Report #1</td>
<td>3</td>
<td>31 January 2018</td>
</tr>
<tr>
<td>Design Review @ NETL</td>
<td>6</td>
<td>04 May 2018</td>
</tr>
<tr>
<td>Host Site Agreement</td>
<td>6</td>
<td>04 May 2018</td>
</tr>
<tr>
<td>Construction, Waste Mgmt Docs for Phys &amp; Chem Pilots</td>
<td>6</td>
<td>04 May 2018</td>
</tr>
<tr>
<td>Quarterly Report #2</td>
<td>6</td>
<td>04 May 2018</td>
</tr>
<tr>
<td>TIM #2 @ WWS (Chem Plant Site Visit)</td>
<td>9</td>
<td>10 August 2018</td>
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<tr>
<td>Quarterly Report #3</td>
<td>9</td>
<td>27 July 2018</td>
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<tr>
<td>Sys Test, Ops, Sample &amp; Analysis Plan for Phys Pilot</td>
<td>10</td>
<td>24 August 2018</td>
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<tr>
<td>TIM #3 @ CAER (Physical Pilot Site Visit/Demo)</td>
<td>12</td>
<td>26 October 2018</td>
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<tr>
<td>Quarterly Report #4</td>
<td>12</td>
<td>29 October 2018</td>
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<tr>
<td>Sys Test, Ops, Sample &amp; Analysis Plan for Chem Pilot</td>
<td>13</td>
<td>30 November 2018</td>
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<tr>
<td>Quarterly Report #5</td>
<td>15</td>
<td>28 January 2019</td>
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<tr>
<td>TIM #4 @ PSI (Micropilot Site Visit)</td>
<td>15</td>
<td>08 February 2019</td>
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<tr>
<td>TIM #5 @ WWS (Chemical Pilot Shakedown Tests)</td>
<td>18</td>
<td>26 April 2019</td>
</tr>
<tr>
<td>Quarterly Report #6</td>
<td>18</td>
<td>29 April 2019</td>
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<tr>
<td>Physical Pilot Test Report</td>
<td>19</td>
<td>04 June 2019</td>
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<tr>
<td>Quarterly Report #7</td>
<td>21</td>
<td>29 July 2019</td>
</tr>
<tr>
<td>TIM #6 @ NETL (Readiness Review)</td>
<td>22</td>
<td>04 September 2019</td>
</tr>
<tr>
<td>Quarterly Report #8</td>
<td>24</td>
<td>28 October 2019</td>
</tr>
<tr>
<td>TIM #7 @ WWS (Chemical Pilot Demo)</td>
<td>26</td>
<td>06 January 2020</td>
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<tr>
<td>Quarterly Report #9</td>
<td>27</td>
<td>31 January 2020</td>
</tr>
<tr>
<td>Final REYSce Product Split Sample Delivery</td>
<td>27</td>
<td>07 February 2020</td>
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<tr>
<td>Chemical Pilot Test Report</td>
<td>28</td>
<td>28 February 2020</td>
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<tr>
<td>Techno-Economic Analysis Report</td>
<td>28</td>
<td>28 February 2020</td>
</tr>
<tr>
<td>Technology Development and Commercial Plant Design</td>
<td>28</td>
<td>28 February 2020</td>
</tr>
<tr>
<td>Phase 2 Final Report</td>
<td>30</td>
<td>30 April 2020</td>
</tr>
<tr>
<td>Phase 2 Final Briefing @ NETL</td>
<td>30</td>
<td>30 April 2020</td>
</tr>
</tbody>
</table>
Summary of Phase 2 Results To Date
## Phase II Status

<table>
<thead>
<tr>
<th>Performance Attributes</th>
<th>Commercial Target Performance Requirements</th>
<th>Current Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feedstock REYSc Content</td>
<td>&gt;300 ppm (whole mass basis)</td>
<td>Feedstock REYSc content &gt;500 ppm has been achieved by CAER.</td>
</tr>
<tr>
<td>Total REYSc content in final concentrate</td>
<td>&gt;10 wt.% (elemental basis)</td>
<td>REYSc final content of 10 – 20 wt.% has been recorded at Micropilot scale. Enrichment at chemical pilot scale TBD.</td>
</tr>
<tr>
<td>Return on Investment</td>
<td>&lt;7 years</td>
<td>Detailed economic forecasts ongoing. Cost and revenue drivers, potential plant locations, and potential suppliers and purchasers identified and quantified.</td>
</tr>
<tr>
<td>Delivered Concentrate Quantity</td>
<td>0.05 kg</td>
<td>Should be readily achievable based on chemical pilot mass balance calculations</td>
</tr>
<tr>
<td>Final REE Yield</td>
<td>&gt;10 wt.%</td>
<td>REYSc yields of 10-15 wt.% recorded in Micropilot. Chemical pilot yields TBD.</td>
</tr>
<tr>
<td>Cement Substitute Yield</td>
<td>&gt;90 wt.%</td>
<td>Consistent cement substitute yields of 90-93 wt.% recorded in the Micropilot. Yield at chemical pilot scale TBD.</td>
</tr>
<tr>
<td>Solvent/ Reagent Recycling</td>
<td>Solvent&gt; 98.5 wt.%, Reagent&gt;90 wt.%</td>
<td>Solvent recovery of ~97 wt.% &amp; reagent recovery of 93 wt.% recorded in Micropilot. Solvent recycling efficiency expected to increase at pilot scale.</td>
</tr>
</tbody>
</table>

At present, PSI expects that all target performance requirements are achievable.
• Ash from eastern KY coal selected for Phase 2 developments

• The average REYSn content of 556 ppm measured from a composite of 20 ash samples > 300 ppm (DOE requirement)

• Example:

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sc</th>
<th>Y</th>
<th>ΣREE</th>
<th>ΣREY</th>
<th>LREE/HREE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composite</td>
<td>33</td>
<td>59</td>
<td>457</td>
<td>516</td>
<td>7.46</td>
</tr>
</tbody>
</table>
Individual Rare Earth Elemental Content for Selected Ash Fraction

Significant content of Nd (~180 ppm), Y (~50 ppm), and Sc (~25 ppm)
Reasonable (~10 ppm) content of Pr, Gd, Dy
PSI Micropilot Facility

PSI Micropilot facility is operational and has demonstrated target yield and enrichment performance requirements.
Micropilot Processing of Selected Ash Fraction

- Developed chemical processes for the selected ash fraction to recover REYSc with high yield and high enrichment in two final concentrate products of commercial value.
  - Concentration expressed on elemental basis i.e. the content of REY or Sc relative to that of all elements)

- **Product 1: REY-rich concentrate:**
  - REY Concentration > 15 wt%
    - REY concentration >> threshold target (10 wt%)
    - REY:Sc 100:1
    - LREE:HREE ratio = 2.3:1

- **Product 2: Sc-rich concentrate:**
  - REY:Sc 6:1

- Process improves LREE:HREE Ratio by 2X in Product 1
  - Starting Coal Ash LREE:HREE Ratio = 5:1
CAER Physical Processing Pilot Plant

CAER physical pilot plant is operational and ~8 tons of coal ash have been processed; ~50% Yield for ash mass fraction for chem processing.
Pozzolanicity Testing – Strength Activity Index

- **Strength Activity Index or SI:** how the coal ash contributes to the strength of concrete.
- Typically measured as the compressive strength of a standard mortar mix with fly ash substituting at a defined level for Portland cement after a defined period of curing (Blue Line).
- SI is then compared as a ratio percent to a mortar with all Portland cement (Green Line).
- ASTM SI criterion is 75% at 7 days or 28 days (Red line). The processed fine ash utilized at 20% replacement of OPC achieved a strength index greater than 75 by 28 days of curing.
WWS Chemical Processing Pilot Plant

- Detailed design completed
- Long lead time equipment has been ordered
- Infrastructure (utilities) installation in progress
- Plant assembly beginning May 2019
- Shakedown to begin July 2019
Techno-economic Modeling Approach – Phase 1

- Chemical processing and economics modeled in Aspen
  - Capital and operating expenses per model
    - Modified per our team’s experience
  - Result: Pro forma spreadsheet model

- Physical processing economics modeled
  - Capital and operating expenses per CAER experience
  - Result: Pro forma spreadsheet model

- Integrated process economics modeled
  - Added capital expenditures of physical and chemical processes

- Modular, transportable physical and chemical processing plants

➢ Phase I Model: AACE Level 3
## AACE Estimate Classes

<table>
<thead>
<tr>
<th>ESTIMATE CLASS</th>
<th>Primary Characteristic</th>
<th>Secondary Characteristic</th>
<th>Secondary Characteristic</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MATURITY LEVEL OF PROJECT DEFINITION DELIVERABLES</td>
<td>END USAGE</td>
<td>METHODOLOGY</td>
</tr>
<tr>
<td></td>
<td>Express as % of complete definition</td>
<td>Typical purpose of estimate</td>
<td>Typical estimating method</td>
</tr>
<tr>
<td>Class 5</td>
<td>0% to 2%</td>
<td>Concept screening</td>
<td>Capacity factored, parametric models, judgment, or analogy</td>
</tr>
<tr>
<td>Class 4</td>
<td>1% to 15%</td>
<td>Study or feasibility</td>
<td>Equipment factored or parametric models</td>
</tr>
<tr>
<td>Class 3</td>
<td>10% to 40%</td>
<td>Budget authorization or control</td>
<td>Semi-detailed unit costs with assembly level line items</td>
</tr>
<tr>
<td>Class 2</td>
<td>30% to 75%</td>
<td>Control or bid/tender</td>
<td>Detailed unit cost with forced detailed take-off</td>
</tr>
<tr>
<td>Class 1</td>
<td>65% to 100%</td>
<td>Check estimate or bid/tender</td>
<td>Detailed unit cost with detailed take-off</td>
</tr>
</tbody>
</table>

Notes: [a] The state of process technology, availability of applicable reference cost data, and many other risks affect the range markedly. The +/- value represents typical percentage variation of actual costs from the cost estimate after application of contingency (typically at a 50% level of confidence) for given scope.
Summary of Phase II Economic Model Updates

- Defined waste streams, calculated daily waste yield and determined handling/disposal costs based on vendor quotes
- Incorporated costs associated with coal ash excavation and transportation of dry nitrate to a chemical plant
- Refined material acquisition costs at scale based on vendor quotes
- Evaluated product transportation methods and associated distance-costs (truck, rail, barge)
- Defined standards for use of ash cake as a Portland cement substitute
- Defined a protocol for screening ash from a given site for PSI process suitability
- Investigated potential markets and customers for REE-rich product. Identified REE-oxides as a potential market entry point

Phase II Model: AACE Level 2

PSI TEA is currently between AACE Class 3 and AACE Class 2 model
Updated Phase 2 Techno-Economic Model

Representative 1200 tpd ash physical processing plant and 600 tpd chemical processing plant

Representative capital expenses (CAPEX)
$49 M

Representative operating expenses (OPEX)
$32M USD/year

Payback period <8 years at 2018 REYSc prices.
Backups
Phase 2 Objective:

- Demonstrate the REYSc separation/enrichment technology developed in Phase 1 in a pilot scale plant with operating capacity of 0.1-1 metric ton per day (tpd)

Specific Objectives:

1. Refine and complete detailed design of the chemical pilot plant(s) from Phase 1
   - Modular, transportable designs

2. Assemble and operate a Micropilot (chemical) plant for quick turnaround validation of pilot plant processing parameters, and provide data for chemical pilot plant design
Phase 2 Project Objectives - II

3. Assemble pilot scale plant at CAER for physical processing of ash

4. Construct a modular pilot scale chemical plant at WWS facilities

5. Demonstrate operation of the physical pilot plant using the power plant (ash feedstock selected in Phase 1)
   - Modular, mobile CAER plant that uses the selected ash feedstock
   - Operation at ash source, decoupled from chemical pilot plant
6. Demonstrate operation of the chemical pilot plant using the selected ash fraction produced by the physical pilot plant
   - Operation at WWS facility in Sharon, PA
     • Selected ash fraction transported to this facility from Lexington, KY

7. Refine and enhance the Phase 1 techno-economic model using results of above physical and chemical pilot plant operations
   - Produce AACE Class 2 costing fidelity model in Phase 2
     • Current Phase 1 model is AACE Class 3

8. Develop and provide design of a commercial scale plant for profitable REYSc recovery from coal ash at Phase 2 conclusion
   - Use the above refined Phase 2 techno-economic model
   - ROI metrics as previously stated